इंटरनेट मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

"जानने का अधिकार, जीने का अधिकार"
Mazdoor Kisan Shakti Sangathan
"The Right to Information, The Right to Live"

"पुराने को छोड नये के तरफ"
Jawaharlal Nehru
"Step Out From the Old to the New"

IS 8072 (1984): Quinalphos, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]

"ज्ञान से एक नये भारत का निर्माण"
Satyanarayan Gangaram Pitroda
"Invent a New India Using Knowledge"

Act Number 22 of 2005
The Indian Parliament
RIGHT TO INFORMATION

Made Available By
Public.Resource.Org

"ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है"
Bhartṛhari—Nītiśatakam
"Knowledge is such a treasure which cannot be stolen"

BLANK PAGE

BRUTUM FULMEN

IGNORANTIA JURIS NON EXCUSAT

PROTECTED BY COPYRIGHT

IS : 8072 - 1984

(Reaffirmed 2012)

Indian Standard
SPECIFICATION FOR QUINALPHOS, TECHNICAL
(First Revision)

(First Reprint DECEMBER 1997)

UDC 632.951 QUINALPHOS

© *Copyright* 1985

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3 *January* 1985

Indian Standard

SPECIFICATION FOR QUINALPHOS, TECHNICAL

(*First Revision*)

Pest Control Sectional Committee, AFCDC 6

Chairman	*Representing*
DR K. D. PAHARIA	Directorate of Plant Protection, Quarantine & Storage (Ministry of Agriculture), Faridabad
Members	
DR P. K. AGGARWAL	IDMA Laboratories, Chandigarh
Da S. R. IYENGAR (*Alternate*)	
DR H. L. BAMI	Pesticides Residue Analysis Sectional Committee, AFDC 56, ISI
DR B. BANERJEE	Tea Research-Association, Jorhat
DR G. SATYANARAYANA (*Alternate* I)	
SHRI S. C. DAS (*Alternate* II)	
SHRI N. S. BIRDIE	Shriram Test House, Delhi
SHRI R. K. BANERJEE (*Alternate*)	
CHIEF PLANT PROTECTION OFFICER	Department of Agriculture, Government of Maharashtra, Bombay
CHEMIST INCHARGE, INSECTICIDES TESTING LABORATORY (*Alternate*)	
DR M. S. DHATT	National Malaria Eradication Programme, Delhi
SHRI V. DORAIRAJ	Plastic Containers Sectional Committee, MCPD 11, IS1
SHRI G. D. GOKHALE	Bombay Chemicals Private Limited, Bombay
SHRI V. V. KETKAR (Alternate)	
SHRI N. L. KALRA	National Institute of Communicable Diseases, Delhi
SHRI G. C. JOSHI (*Alternate*)	
DR R. L. KALRA	Punjab Agricultural University, Ludhiana
DR R. P. CHAWLA (*Alternate*)	
DR KALYAN SINGH	Chander Sekher Azad University of Agriculture & Technology, Kanpur
DR S. K. KASHYAP	National Institute of Occupational Health (ICMR), Ahmadabad
DR S. K. GUPTA (*Alternate*)	
SHRI J. K. KHOSLA	Metal Containers Sectional Committee, MCPD 12, IS1
SHRI A. N. BHATTACHARYYA (*Alternate*)	
SHRI S. G. KRISHNAN	National Organic Chemical Industries Limited, Bombay
DR J. S. VERMA (*Alternate*)	

(*Continued on page 2*)

© *Copyright* 1985
BUREAU OF INDIAN STANDARDS
This publication is protected under the *Indian Copyright Act* (XIV of 1957) and reproduction in whole or in part by any *means* except with written permission of the publisher shall be deemed to be an infringement of copyright under the said Act

(*Continued* from *page* 1)

Members	*Representing*
Dr S. Lakshmanan	Paper and Flexible Packaging Sectional Committee, MCPD 14. ISI
Dr J. C. Majumdar	BASF India Limited, Bombay
Dr B. P. Chandrasekhar (*Alternate*)	
Shri S. K. Majumdar	Central Food Technological Research Institute (CSIR), Mysore
Shri M. Muthu (*Alternate*)	
Shri K. S. Mehta	Bharat Pulverising Mills Private Limited, Bombay
Shri S. Chatterjee (*Alternate*)	
Shri L. S. Mirle	Agromore Limited, Bangalore
Shri S. K. Raman (*Alternate*)	
Shri J. M. Modi	Pesticides Formulators Association of India (S.S.I.), Bombay
Dr S. R. Barooah (*Alternate*)	
Dr S. K. Mukerjee	Agricultural Chemicals Division, Indian Agricultural Research Institute (ICAR), New Delhi
Dr N. K. Roy (*Alternate*)	
Shri K. Rajendran Nair	Central Insecticides Cell, Directorate of Plant Protection, Quarantine & Storage (Ministry of Agriculture), Faridabad
Dr R. C. Gupta (*Alternate* I)	
Shri S. K. Ghosh (*Alternate* II)	
Shri P. V. Narayanan	Indian Institute of Packaging, Bombay
Shri K. B. Gwta (*Alternate*)	
Dr V. N. Nigam	Ministry of Defence (DGI)
Shri M. M. Padalia	Department of Agriculture, *Government* of Gujarat, Ahmadabad
Shri R. G. Jadbja (*Alternate*)	
Shri A. R. Panicker	Hindustan Insecticides Limited, New Delhi
Dr S. N. Deshmukh (*Alternate*)	
Shri Y. A. Pradhan	Rallis India Limited, Bombay
Shri M. L. Shah (*Alternate*)	
Dr R. L. Rajak	Central Insecticides Laboratory, Directorate of Plant Protection, Quarantine & Storage (Ministry of Agriculture), Faridabad
Shri V. C. Bhargava (*Alternate* I)	
Deputy Director (PR & QC) (*Alternate* II)	
Representative	Department of Agriculture, Government of Andhra Pradesh, Hyderabad
Dr K. D. Sharma	Directorate General of Technical Development, New Delhi
Shri S. C. Bajaj (*Alternate*)	
Dr K. N. Shrivastava	Pesticides Association of India, New Delhi
Dr J. C. Majumdar (*Alternate*)	
Dr K. N. Shrivastava	Shaw Wallace & Co Limited, Calcutta
Shri R. N. Roychowdhury (*Alternate*)	
Shri A C Shroff	Excel Industries Limited, Bombay
Shri P. V. Kango (*Alternate*)	
Dr K. Sivasankaran	Union Carbide (India) Limited, New Delhi
Dr U. V. Singh (*Alternate*)	

(*Continued on page* 10)

Indian Standard

SPECIFICATION FOR QUINALPHOS, TECHNICAL

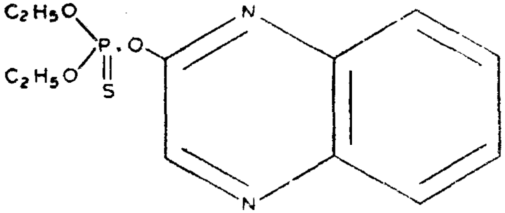
(***First*** *Revision*)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 29 October 1984, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Quinalphos is the accepted common name by the International Organization for Standardization (ISO) for *OO*-quinoxalin-2-yl phosphorothioate. The empirical and structural formulae and molecular mass are as given below:

Empirical Formula	*Structural Formula*	*Molecular Mass*
$C_{12}H_{15}O_3N_2PS$		298·3



0.3 This standard was first published in 1976 and an amendment was issued to it. This revision incorporates a thin layer chromatography method followed by ultraviolet absorption procedure for determination of purity of quinalphos.

0.4 In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides* Act, 1968 and Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for quinalphos, technical.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of a reddish brown to dark brown clear liquid free from extraneous matter.

'2.2 The material shall also comply with the requirement specified in Table 1.

TABLE 1 REQUIREMENTS FOR QUINALPHOS, TECHNICAL

SL No	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO	
			Appendix of this Standard	Cl No. of IS : 6940-1982*
(1)	(2)	(3)	(4)	(5)
i)	Quinalphos content, percent by mass, *Min*	70·0	A	—
ii)	Water content, percent by mass, *Max*	0·1	—	4
iii)	Relative density at 25/25°C, *Min*	1·04		5
iv)	Material insoluble in acetone, percent by mass, *Max*	0·1	—	9
v)	Acidity (as H_2SO_4), percent by mass, *Max*	0·4	—	11·3

*Methods of test for pesticides and their formulations (*first revision*).

3. PACKING AND MARKING

3.1 Packing — The material shall be packed as per requirements given in IS : 8190 (Part 2)-1980†.

---

*Rules for rounding off numerical values (revised).

†Requirements for packing of pesticides: Part 2 Liquid pesticides (first *revision*).

3.2 Marking — The container shall bear legibly and indelibly the following information and any other information as is necessary under the ***Insecticides Act*** and Rules:

a) Name of the material;
b) Name of the manufacturer;
c) Batch number;
d) Date of manufacture;
e) Net volume of contents;
f) Quinalphos content, percent (*m/m*); and
g) Cautionary notice worded as in ***Insecticides Act*** and Rules,

3.2.1 Each container may also be marked with the Standard Mark.

3.2.2 The use of the Standard Mark is governed by the provisions of Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 Representative samples of the material shall be drawn in accordance with IS : 10946-1984*

5. TESTS

5.1 Tests shall be carried out as prescribed in col 4 and 5 of Table 1.

5.2 **Quality of Reagents** — Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1977†) shall be employed in tests.

NOTE — ‘ Pure chemicals ’ shall mean chemicals that do not contain impurities which affect the result of an analysis.

---

*Methods for sampling of technical grade pesticides.

†Specification for water for general laboratory use (*second* revision)

APPENDIX A

[Table 1, *Item* (i)]

DETERMINATION OF QUINALPHOS CONTENT

A-0. GENERAL

A-0.1 Two methods, namely, thin layer chromatography followed by UV absorption or volumetric method may be used for determining active ingredient content. However, in case of dispute thin layer chromatographic method shall be the referee method.

A-X. THIN LAYER CHROMATOGRAPHIC METHOD

A-1.1 Principle — Quinalphos is separated from associated impurities in case of technical grade material and from auxiliary material in case of formulations and its concentration is measured spectrophotometrically using pure quinalphos of known purity. The absorbance is measured in the ultraviolet region at 237 and 320 nm.

A-1.2 **Apparatus**

A-1.2.1 *Spectrophotometer* — capable of recording or measuring in the region of 220 to 320 nm.

A-1.2.2 *Coated Glass Plates with Silica Gel-G Containing Flourescent Indicator 254 nm — of* size 20 x 20 cm and layer thickness of 0·25 mm.

A-1.2.3 *TLC Tank* — to accommodate TLC plates of 20 x 20 cm.

A-1.2.4 *Filter Paper* — Whatman No. 1 or equivalent.

A-1.2.5 *Micro-pipette* — 5-µl or 10-µl capacity.

A-1.2.6 *Spatula* — stainless steel.

A-1.2.7 *Centrifuge* — with 15-ml capacity stoppered centrifuge tubes.

A-1.2.8 *Volumetric Flasks* — 25-ml capacity.

A-1.2.9 *UV Lamp* — 254 nm.

A-1.2.10 *Burette* — Class A.

A-1.2.11 *Plastic Marker* — with $(2{\cdot}5)^2$ cm slot.

A-1.3 Reagents

A-1.3.1 *Toluene* — LR grade.

A-1.3.2 *Ethyl Acetate* — LR grade.

A-1.3.3 *Methanol* — spectroscopy grade.

A-1.3.4 *Quinalphos — of* plus 99 percent purity. To be purified by column chromatography everytime an analysis is performed.

A-I.4 **Preparation of Solvent System**

A-1.4.1 Mix 95 parts of toluene with 5 parts of ethyl-acetate.

A-l.5 **Preparation of Standard and Sample Sloutions**

A-1.5.1 *Preparation of Standard Solution* — Weigh accurately 0·5 g of standard quinalphos (*see* A-1.3.4) into 25-ml volumetric flask and make up to the mark with toluene.

A-1.5.2 *Preparation of Sample Solution* — Weigh accurately the quantity of sample to contain 0·5 g of quinalphos into 25-ml volumetric flask and make up to the mark with toluene.

A-l.6 Procedure

A-1.6.1 *Spotting and Loading* — Mark 6 positions on **TLC** plate equidistantly and horizontally with pencil which should fall at about 3 cm away from the edge. These are the starting points. Leave first mark as ' Blank '. At second position spot 5 µl of standard solution and at remaining four 5 µl of sample solution.

A-1.6.2 Place about 150 to 200 ml of solvent system in TLC tank which already contains a filter paper sticking to it internally. Allow the chamber to saturate for about to 30 minutes.

A-1.6.2.1 Place the spotted plate into the chamber and run the solvent system to a height of 14 cm.

A-1.6.2.2 Remove the plate and allow. it to dry.

.A-1.6.2.3 Place the plate under 254 nm UV-lamp and encircle the quinalphos spots with pencil.

A-1.6.2.4 To have exact areas of spots, square mark with a plastic marker all the spots with pencil. Also square-mark 'Blank' falling in line with quinalphos spots horizontally.

A-1.6.3 *Scooping* — First scoop out the 'Blank' spot carefully on a piece of butter paper and transfer quantitatively to centrifuge tube. Similarly scoop out successive spots individually and transfer quantitatively to centrifuge tubes.

A-1.6.3.1 Add accurately 10 ml of methanol (see A-1.3.3) from the burette to each tube and shake gently for 5 minutes.5

A-1.6.3.2 Centrifuge these solutions to get a clear supernatant layer.

A-1.6.4 ***Absorption Measurement*** — Measure the absorbance of these solutions at 237 nm or at 320 nm using methanol as reference blank

A-1.6.5 ***Calculation***

Quinalphos content, percent by mass $= \frac{A}{B} \times \frac{M}{M_1} \times P$

where

A = absorbance of sample;
M = mass, in g, of standard;
P = percent purity of reference standard;
B = absorbance of standard; and
M = mass, in g, of sample.

NOTE -- Under the prescribed conditions, for 100 percent pure quinalphos, the following absorbance values are obtained at:

237 nm	**0.786**
320 nm	**0.209**

A-2. VOLUMETRIC METHOD

A-2.1 Principle — Quinalphos is decomposed by heating it with hydrogen peroxide and concentrated sulphuric acid, and the phosphate obtained is estimated volumetrically by the molybdate method.

A.2.2 Reagents

A-2.2.1 ***Nitric Acid — 70*** percent.

A-2.2.2 ***Sulphuric Acid — 96*** percent.

A-2.2.3 ***Citric-Molybdate Reagent*** — prepared as in A-2.2.3.1 and A-2.2.3.2.

A-2.2.3.1 To ***54 g*** anhydrous molybdic acid, add 200 ml water and 15 g sodium hydroxide. Heat and stir until dissolved.

A-2.2.3.2 Dissolve 60 g citric acid in 250 to 300 ml water. Add 140 ml concentrated hydrochloric acid (*r d* = 1·18). To this solution, add solution prepared in A-2.2.3.1 with stirring. Filter and dilute to one litre. This solution gives blue-green tinge. Add 0·5 to 1 percent potassium bromate till the colour becomes yellow.

A-2.2.4 ***Hydrogen Peroxide — 30*** percent (*v/v*).

A-2.2.5 ***Mixed Indicator Solution*** — prepared by mixing 3 volumes of solution I and 2 volumes of solution- II.

Solution I — Dissolve 0·1 g thymol blue in 2·2 ml of 0·1 N sodium hydroxide and 50 ml alcohol. Dilute to 100 ml with water.

Solution II — Dissolve 0·1 g phenolphthalein in 60 ml alcohol and dilute to 100 ml with water.

A-2.2.6 *Quinoline Solution* — Heat 60 ml concentrated hydrochloric acid in 350 to 400 ml water to 70°C to 80°C. To this hot solution add 50 g quinoline, stir and after cooling, dilute to 1 litre.

A-2.2.7 *Hydrochloric Acid* — concentrated and 0·5 N.

A-2.2.8 *Sodium Hydroxide Solution* — 0·5 *N.*

A-2.3 Procedure

A-2.3.1 Weigh accurately sufficient amount of sample to contain approximately 0·5 g quinalphos in a glass ampoule and introduce it in a 500-ml Khjedhal digestion flask. Add 20 ml of hydrogen peroxide and cool in ice cold water. Add 10 ml of sulphuric acid and heat gently under controlled conditions till white fumes of sulphur trioxide come out. Allow to cool, add 15 ml of nitric acid and heat again to disappearance of brown fumes and appearance of white fumes of sulphur trioxide. Allow to cool, add 20 ml of hydrogen peroxide and heat again till white fumes are given off. Cool and make up to 500 ml.

A-2.3.2 To 100 ml aliquot from A-2.3.1 add 50 ml of citric molybdate reagent. Heat to boiling and add very slowly, dropwise, 25 ml of quinoline solution with constant mechanical stirring. Boil for 5 minutes more, cool and filter the precipitate through a gooch crucible. Wash with water till free from acid. Dissolve the precipitate in a known excess of sodium hydroxide solution (about 50 ml of 0·5 N). Titrate excess alkali with 0 5 N hydrochloric acid using mixed indicator. Carry out a blank determination on the reagents and subtract from the volume consumed by the sample.

A-2.4 Calculation

$$\text{Quinalphos content, percent by mass} = \frac{v}{m} \times 2{\cdot}867$$

where

v = volume, in ml, of standard sodiu'm hydroxide solution; and

m = mass, in g, of sample taken for the test.

(Continued from page 2)

Members	*Representing*
SHRI *N. S.* VENRATARAMAN	Department of Agriculture, Government of Tamil Nadu, Madras
DR R. V. VENKATARATNAM DR NAGABHUSHAN RAO (*Alternate*)	Regional Research Laboratory (CSIR), Hyderabad
DR SANTOSH YELLORE DR D. B. ANANTRA NARAYANA (*Alternate*)	Analytical Testing Services Pvt Ltd, New Delhi
SHRI T. PURNANANDAM, Director (Agri & Food)	Director General, IS1 (*Ex-officio Member)*

Secretary

SHRI LAJINDER SINGH
Deputy Director (Agri & Food), IS1

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002
Telephones: 323 0131, 323 3375, 323 9402
Fax : 91 11 3234062, 91 11 3239399, 91 11 3239382

Telegrams : Manaksanstha
(Common to all Offices)

Central Laboratory :

	Telephone
Plot No. 20/9, Site IV, Sahibabad Industrial Area, Sahibabad 201010	8-77 00 32

Regional Offices:

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002	323 76 17
*Eastern : 1/14 CIT Scheme VII M, V.I.P. Road, Maniktola, CALCUTTA 700054	337 86 62
Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160022	60 38 43
Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600113	235 23 15
†Western : Manakalaya, E9, Behind Marol Telephone Exchange, Andheri (East), MUMBAI 400093	832 92 95

Branch Offices::

'Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMEDABAD 380001	550 13 48
‡Peenya Industrial Area, 1st Stage, Bangalore-Tumkur Road, BANGALORE 560058	839 49 55
Gangotri Complex, 5th Floor, Bhadbhada Road, T.T. Nagar, BHOPAL 462003	55 40 21
Plot No. 62-63, Unit VI, Ganga Nagar, BHUBANESHWAR 751001	40 36 27
Kalaikathir Buildings, 670 Avinashi Road, COIMBATORE 641037	21 01 41
Plot No. 43, Sector 16 A, Mathura Road, FARIDABAD 121001	8-28 88 01
Savitri Complex, 116 G.T. Road, GHAZIABAD 201001	8-71 19 96
53/5 Ward No.29, R.G. Barua Road, 5th By-lane, GUWAHATI 781003	54 11 37
5-8-56C, L.N. Gupta Marg, Nampally Station Road, HYDERABAD 500001	20 10 83
E-52, Chitaranjan Marg, C-Scheme, JAIPUR 302001	37 29 25
117/418 B, Sarvodaya Nagar, KANPUR 208005	21 68 76
Seth Bhawan, 2nd Floor, Behind Leela Cinema, Naval Kishore Road, LUCKNOW 226001	23 89 23
NIT Building, Second Floor, Gokulpat Market, NAGPUR 440010	52 51 71
Patliputra Industrial Estate, PATNA 800013	26 23 05
Institution of Engineers (India) Building 1332 Shivaji Nagar, PUNE 411005	32 36 35
T.C. No. 14/1421, University P. O. Palayam, THIRUVANANTHAPURAM 695034	621 17

---

*Sales Office is at 5 Chowringhee Approach, P.O. Princep Street, CALCUTTA 700072	27 10 85
†Sales Office is at Novelty Chambers, Grant Road, MUMBAI 400007	309 65 28
‡Sales Office is at **'F' Block, Unity Building, Narashimaraja Square, BANGALORE 560002**	222 39 71

Printed at Dee Kay Printers, New Delhi-110015, India.

AMENDMENT NO 1 JULY 1987
TO
IS 8072-1984 SPECIFICATION FOR QUINALPHOS, TECHNICAL
(First Revision)

(***Pages*** 6, 7 ***and*** 8, ***clauses*** A-0 to A-1.6.5) — Substitute the following for the existing matter:

‘A-0. GENERAL

A-0.1 Two methods, namely, gas liquid chromatographic (GLC) or volumetric method may be used for determining active ingredient content. However, in case of dispute GLC method shall be the referee method.

A-1. GAS LIQUID CHROMATOGRAPHIC METHOD

A-1.1 Principle — The quinalphos content is determined using an internal standard on a GLC equipped with a FID detector and integrator.

A-1.2 Apparatus

A-1.2.1 ***Gas Liquid Chromatograph*** — A gas chromatograph equipped with a FID detector is operated under the following suggested parameters. These parameters may be varied as per available facilities provided standardization is done.

Column	Glass 2 metres long and 3 mm i.d. packed with 3 percent OV-17 on 80 to 100 mesh gas chrom Q
Column oven temperature	210°C
Injection port temperature	250°C
Detector temperature	250°C
Gas flow rate	Nitrogen, 30 ml/min

A-1.2.2 ***Micro-Syringe*** — 2-µl capacity.

A-1.3 Reagents

A-1.3.1 ***Standard Reference Quinalphos*** — of known purity.

A-1.3.2 ***Acetone/Toluene***

A-1.3.3 Internal Standard — Di-n-butyl phthalate.

A-1.4 Procedure

A-1.4.1 ***Preparation of Internal Standard*** (***I_S***) ***Solution*** — Weigh accurately 1’5 g of di-n-butyl phthalate in a 50-ml volumetric flask and make up to volume using acetone/toluene. This will give a solution containing 30 mg/ml of I_S.

A-1.4.2 ***Preparation of Standard Solution*** — Weigh **accurately** equivalent to **0'2** *g* of quinalphos in a beaker. Add 5 ml of I_s (see A-1.4.1). Shake well to dissolve and transfer carefully to a **25-ml** volumetric flask and make up the volume with acetone/toluene.

A-1.4.3 ***Preparation of Sample Solution*** — Weigh out a sample containing equivalent to 0’2 g of quinalphos and proceed as per A-1.4.2.

A-1.4.4 ***Analysis of the Sample*** — Inject 2 µl of the standard and, if necessary, adjust the parameters and the column injected to give a complete separation within a reasonable time and peak heights half to three fourths full scale. Proceed with determination at least re-injecting each of standard and sample solution in random order.

A-1.4.5 Measure the peak heights of areas of quinalphos and internal standard solution. Determine the R_I values for each injected **standard** — internal standard solution.

A-l.5 Calculation

$$\text{Quinalphos content, percent by mass} = \frac{MI_s \times A_s}{M_s \times AI_s} \times 100 \times R_t$$

where

MI_s = mass, in g, of the internal standard;
A_s = area of the sample peak;
R_t = response factor $= \dfrac{M_s \times AI_s}{M_s \times A_s}$
M_s = mass, in g, of the sample taken; and
AI_s = area of the internal standard peak.

(AFCDC 6)

Printed at Dee Kay Printers, New Delhi-l 10015, India.

AMENDMENT NO. 2 MARCH 1989

TO

IS : 8072 - 1984 SPECIFICATION FOR QUINALPHOS, TECHNICAL

(*First Revision*)

[*Page* 6, *clause* A-O.1 (see also Amendment No. 1)] — Substitute the following for the existing clause:

'A-O.1 Two methods, namely, gas liquid chromatographic (GLC) or thin layer chromatography followed by UV absorption may be used for determining active ingredient content. However, in case of dispute GLC method shall be the referee method.'

(*Page* 2, Amendment No. 1) — Insert the following matter after A-1.5:

"A-2. THIN LAYER **CHROMATOGRAPHIC** METHOD

A-2.1 Principle — Quinalphos is separated from associated impurities in case of technical grade material and from auxiliary material in case of formulations and its concentration is measured spectrophotometrically using pure quinalphos of known purity. The absorbance is measured in the ultraviolet region at 237 and 320 nm.

A-2.2 Apparatus

A-2.2.1 *Spectrophotometer* — Capable of recording or measuring in the region of 220 to 320 nm.

A-2.2.2 *Coated Glass Plates with Silica Gel G Containing Fluorescent Indicator 254 nm* — *of* size *20* × *20* cm and layer thickness of 0·25 mm.

A-2.2.3 *TLC Tank* — To accommodate TLC plates of 20 x 20 cm.

A-2.2.4 *Filter Paper* — Whatman No. 1 or equivalent.

A-2.2.5 *Micro-pipette* — 5-µl or 10-µl capacity

A-2.2.6 *Spatula* — Stainless steel.

A-2.2.1 *Centrifuge* — with 15-ml capacity stoppered centrifuge tubes.

A-2.2.8 *Volumetric Flasks* — 25-ml capacity.

A-2.2.9 *UV Lamp* — *254* nm.

A-2.2.10 *Burette* — *Class* A.

A-2.2.11 Plastic Marker — with (2·5)^2 cm slot.

A-2.3 Reagents

A-2.3.1 ***Toluene*** — AR grade.

A-2.3.2 ***Ethyl Acetate*** — AR grade.

A-2.3.3 Methanol — Spectroscopy grade.

A-2.3.4 ***Quinalphos — of*** known purity.

A-2.4 Preparation of Solvent System

A-2.4.1 Mix 95 parts of toluene with 5 parts of ethyl-acetate.

A-2.5 Preparation of Standard and Sample Solutions

A-2.5.1 ***Preparation of Standard Solution*** — Weigh accurately 0·5 g of standard quinalphos (see A-2.3.4) into 25-ml volumetric flask and make up to the mark with toluene.

A-2.5.2 ***Preparation of Sample Solution*** — Weigh accurately the quantity of sample to contain 0·5 g of quinalphos into 25-ml volumetric flask and make up to the mark with toluene.

A-2.6 Procedure

A-2.6.1 ***Spotting and Loading*** — Mark 6 positions on TLC plate equidistantly and horizontally with pencil which should fall at about 3 cm away from the edge. These are the starting points. Leave first mark as ' Blank '. At second position spot 5 µl of standard solution and at remaining four 5 µl of sample solution.

A-2.6.2 Place about 150 to 200 ml of solvent system in TLC tank which already contains a filter paper sticking to it internally. Allow the chamber to saturate for about 30 minutes.

A-2.6.2.1 Place the spotted plate into the chamber and run the solvent system to a height of 14 cm.

A-2.6.2.2 Remove the plate and allow it to dry.

A.2.6.2.3 Place the plate under 254 nm UV-lamp and encircle the quinalphos spots with pencil.

A-2.6.2.4 To have exact areas of spots, square mark with a plastic marker all the spots with pencil. Also square-mark ' Blank ' falling in line with quinalphos spots horizontally.

A-2.6.3 ***Scooping*** — First scoop out the ' Blank ' spot carefully on a piece of butter paper and transfer quantitatively to centrifuge tube. Similarly scoop out successive spots individually and transfer quantitatively to centrifuge tubes.

A-2.6.3.1 Add accurately 10 ml of methanol (see A-2.3.3) from the burette to each tube and shake gently for 5 minutes.

A-2.6.3.2 Centrifuge these solutions to get a clear supernatant layer.

A-2.6.4 *Absorption Measurement* — Measure the absorbance of these solutions at 237 nm or at 320 nm using methanol as reference blank.

A-2.6.5 ***Calculation***

$$\text{Quinalphos content, percent by mass} = \frac{A}{B} \times \frac{M}{M_1} \times P$$

where

A = absorbance of sample;

M = mass, in g, of standard;

P = percent purity of reference standard;

B = absorbance of standard; and

M_1 = mass, in g, of sample.

NOTE — **Quinalphos exhibits 2 absorption maxima in the near UV region at 317 and 237 nm, However, the UV measurement at 237 nm is extremely sensitive to impurities eluted from TLC plate as well as to traces of developing solvents. It is, therefore, recommended to use the maximum at 317 nm for routine analysis. The maximum read at 237 nm corrected by the baseline at 400 nm, can be used for confirmation assay.”**

(AFCDC 6)

Printed at Dee Kay Printers. New Delhi-l 10015, India.

AMENDMENT NO. 3 AUGUST 1997
TO
IS 8072 : 1984 SPECIFICATION FOR QUINALPHOS, TECHNICAL

(First Revbion)

(*Page 4, clause 3.1*) — Substitute 'IS 8190 (Part 2) : 1988†' *for* '*IS* : 8190 (Part 2) - 1980†'.

(*Page 4, footnote marked* '†') — Substitute '(second *revision*)' *for* '(*first revision*)' at the end of the title.

(***Page*** 5, ***clause*** 4.1) — Substitute 'IS **10946:1996***' *for* '*IS* : 10946 - 1984*'.

(*Page* 5, *footnote marked* '*') — Insert '(first *revision*)' at **the end** of the text.

(*Page 5, clause* 5.2) -Substitute 'IS 1070 : **1992**†' *for* 'IS : 1070 - 19775'.

(*Page 5. footnote marked* '†') — Substitute 'Reagent grade water (*third revision*)' for the existing title.

[*Page* 6, *Appendix A (see also Amendments No.* 1 *and 2)*] — Substitute the following for the existing matter:

'APPENDIX A

[Table 1, *Item* (i)]

DETERMINATION OF QUINALPHOS CONTENT

A-O GENERAL

Three methods, namely, high performance liquid chromatography (HPLC) or gas liquid chromatography (GLC) or thin layer chromatography (TLC) followed by ultra-violet (UV) spectrophotometry may be used for determination of quinalphos content. However, either the HPLC or GLC shall be the referee methods. In addition, a volumetric method may be used **only** by a manufacturer solely for production quality control purposes. The volumetric method **shall** not be used by an independent testing laboratory.

A-l HPLC METHOD

A-l.1 Principle

Equal volumes of solutions containing known amounts of quinalphos standard and internal standard are injected into the HPLC unit fitted with a UV detector and the response factor (R.F.) is determined. Same volume of solution containing known amounts of the sample and internal standard is injected subsequently into the HPLC unit and quantity of quinalphos is computed by the standard relationship.

A-1.2 Apparatus

A-1.2.1 ***High Performance Liquid Chromatograph***

Equipped with a printer-plotter-cum-integrator and UV detector. The suggestive operative parameters are given below, which can be varied provided standardization is done:

Column	Stainless steel, 25 cm length and 4.6 mm ID; packed with silica of 10 pm particle size
Detector	***W*** (305 nm)
Solvent system	Carbon tetrachloride, 85 percent (v/v) and Chloroform, 15 percent (v/v)
Solvent flow rate	1 .O ml/minute
Sample size	10 µl

A-1.2.2 ***Micro-Syringe***

A-l.3 Reagents

A-1.3.1 ***Internal Standard Solution***

1 g meta phenoxy benzaldehyde (MPB), AR grade, dissolved in 100 ml of the solvent system as described above (see A-1.2.1) in a 100-ml volumeric flask. The final solution will contain 10 mg/ml of internal standard (MPB).

A-1.3.2 ***Quinalphos Standard Solution***

Dissolve 0.1 g quinalphos, reference standard in a 100-ml volumetric flask in the solvent system. Then add 10 ml internal standard solution and finally make up the volume with the solvent system. The final solution will contain 1 000 parts per million of quinalphos and MPB (Solution I).

A-1.3.3 *Sample Solution*

Dissolve in a 100-ml volumetric flask 0.12 g of quinalphos, technical or 0.4 g of quinalphos 25 percent EC sample in the solvent system. Add 10 ml internal standard solution and finally make up the volume with the solvent system.

A-1.4 Estimation

A-1.4.1 Introduce simultaneously 10 µl each of the internal standard solution and quinalphos standard solution into the HPLC unit and calculate the response factor (R.F.).

$$\text{R.F.} = \frac{\text{Peak area for quinalphos}}{\text{Peak area for MPB}} \times \frac{\text{Mass of MPB}}{\text{Mass of quinalphos}} \times \text{Percent purity of quinalphos standard}$$

A-1.4.2 Introduce 10 µl of sample solution into the HPLC unit, measure the peak areas for the internal standard and quinalphos in the sample solution. Calculate the quinalphos content.

A-1.5 Calculation

$$\text{Quinalphos content, percent by mass} = \frac{\text{Peak area for quinalphos}}{\text{Peak area for MPB}} \times \frac{\text{Mass of MPB}}{\text{Mass of sample}} \times 100$$

A-2 GLC METHOD

A-2.1 Principle

The quinalphos content is determined using an internal standard on a GLC equipped with a flame ionization detector (FID) and integrator.

A-2.2 Apparatus

A-2.2.1 *Gas Liquid Chromatograph*

A gas chromatograph equipped with a FID detector is operated under the following suggested parameters. These parameters may be varied as per available facilities provided standardization is done:

Column	Glass, 200 cm long and 3 mm i.d. packed with 3 percent OV-17 on 80 to 100 mesh gas chrom Q
Column oven temperature	210°C

Injection port temperature	250°C
Detector temperature	250°C
Gas flow rate	Nitrogen, 30 ml/min

A-2.2.2 *Micro-Syringe* — *2* µl.

A-2.3 **Reagents**

A-2.3.1 *Quinalphos Reference Standard* -of known purity.

A-2.3.2 *Acetone/Toluene*

A-2.3.3 Internal Standard — Di-n-butyl phthalate.

A-2.4 Procedure

A-2.4.1 *Preparation of Internal Standard Solution*

Weigh 1.5 g of di-n-butyl phthalate in a 50-ml volumetric flask and make up to volume using acetone/toluene. This will give a solution containing 30 mg/ml of internal standard.

A-2.4.2 *Preparation of Reference Standard Solution*

Weigh equivalent to 0.2 g of quinalphos in a beaker. Add 5 ml of internal standard solution (see A-2.4.1). Shake well to dissolve and transfer carefully to a 25-ml volumetric flask and make up the volume with acetone/toluene.

A-2.4.3 *Preparation of Sample Solution*

Weigh a sample containing equivalent to 0.2 g of quinalphos and proceed as described in A-2.4.2.

A-2.4.4 *Estimation*

Inject 2 µl of the standard into the GLC and, if necessary, adjust the parameters and the column injected to give a complete separation within a reasonable time and peak heights half to three-fourths full scale. Proceed with determination at least reinjecting each of standard and sample solution in random order.

A-2.4.5 Measure the peak heights of areas of quinalphos and internal standard solution. Determine the R.F. values for each injected standard and internal standard solution.

A-2.5 Calculation

Quinalphos content, percent by mass = $\frac{MI_s \times A_s}{M_s \times AI_s} \times R_f \times 100$

where

MI_s = mass, in g, of the internal standard;

AS = area of the sample peak;

R_f = response factor = $\frac{M_s \times AI_s}{M_s \times A_s}$;

M_s = mass, in g, of the sample taken; and

AI_s = area of the internal standard peak.

A-3 TLC FOLLOWED BY UV SPECTROPHOTOMETRY

A-3.1 Principle

Quinalphos is separated from associated impurities in case of technical grade material and from auxiliary material in case of formulations and its concentration is measured spectrophotometrically using pure quinalphos of known purity. The absorbance is measured in the ultraviolet region at 237 and 320 nm.

A-3.2 Apparatus

A-3.2.1 *Spectrophotometer*

Capable of recording or measuring in the region of 220 to 320 nm.

A-3.2.2 ***Coated Glass Plates with Silica Gel G Containing Fluorescent Indicator 254 mm*** — of size *20* cm x *20* cm and layer thickness of 0.25 mm.

A-3.2.3 *TLC Tank* — To accommodate TLC plates of 20 cm x 20 cm.

A-3.2.4 *Filter Paper* — **Whatman** No. 1 or equivalent.

A-3.2.5 *Micro-Pippette* — *5* µl or 10 µl.

A-3.2.6 *Spatula* — Stainless steel.

A-3.2.7 *Centrifuge -with* 15 ml capacity stoppered centrifuge tubes.

A-3.2.8 *Volumetric Flasks* — *25* ml.

A-3.2.9 *UV Lamp — 254* nm.

A-3.2.10 Burette — Glass A.

A-3.2.11 *Plastic Marker -with* $(2.5)^2$ cm slot.

A-3.3 Reagents

A-3.3.1 *Toluene -AR* grade.

A-3.3.2 *Ethyl Acetate — AR* grade.

A-3.3.3 *Methanol* — Spectroscopy grade.

A-3.3.4 *Quinalphos Standard — of known* **purity.**

A-3.4 Solvent System

Mix 95 parts of toluene with 5 parts of ethyl acetate.

A-3.5 Preparation of Standard **and Sample Solutions**

A-3.5.1 *Preparation of Standard Solution*

Weigh 0.5 g of standard quinalphos (see A-3.3.4) into a 25-ml volumetric flask and make up to the mark with toluene.

A-3.5.2 *Preparation of Sample Solution*

Weigh the quantity of sample to contain 0.5 g of quinalphos into 25-ml volumetric flask and make up to the mark with toluene.

A-3.6 Procedure

A-3.6.1 *Spotting and Loading*

Mark 6 positions on TLC plate equidistantly and horizontally with pencil which should fall at about 3 cm away from the edge. These are the starting points. Leave first mark as ‘Blank’. At second position spot 5 µl of standard solution and a remaining, four, 5 µl of sample solution.

A-3.6.2 Place about 150 to 200 ml of solvent system in TLC tank which already contains a filter paper sticking to it internally Allow the chamber to saturate for about 30 minutes.

A-3.6.2.1 Place the spotted plate into the chamber and run the solvent system to a height of 14 cm.

A-3.6.2.2 Remove the plate and allow it to dry.

A-3.6.2.3 Place the plate under 254 nm UV lamp and encircle the quinalphos spots with pencil.

A-3.6.2.4 To have exact areas of spots, square mark with a plastic marker all the spots with pencil. Also square mark 'Blank' falling in line with quinalphos spots horizontally.

A-3.6.3 *Scooping*

First scoop out the 'Blank' spot carefully on a piece of butter paper and transfer quantitatively to centrifuge tube. Similarly scoop out successive spots individually and transfer quantitatively to centrifuge tube.

A-3.6.3.1 Add accurately 10 ml of methanol (see A-3.3.3) from the burette to each tube and shake gently for 5 minutes.

A-3.6.3.2 Centrifuge these solutions to get a clear supernatant layer.

A-3.6.4 *Absorption Measurement*

Measure the absorbance of these solutions at 237 nm or at 230 nm using methanol as reference blank.

A-3.7 **Calculation**

Quinalphos content, percent by mass $= \frac{A}{B} \times \frac{M}{M_1} \times P$

where

A = absorbance of sample;

M = mass, in g, of standard;

P = pecent purity of reference standard;

B = absorbance of standard; and

M_1 = mass, in g, of sample.

NOTE — Quinalphos exhibits 2 absorption maxima in the near ***W*** region at 317 and 237 nm. However, the ***W*** measurement at 237 nm is extremely sensitive to impurities eluted from TLC plate as well as to traces of developing solvents. It is, therefore, recommended to use the maximum at 317 nm for routine analysis. The maximum read at 237 nm corrected by the baseline at 400 nm, can be used for confirmation assay.

A-4 VOLUMETRIC METHOD

A-4.1 Principle

Quinalphos is decomposed by heating it with hydrogen peroxide and concentrated sulphuric acid, and the phosphate obtained is estimated volumetrically by the molybdate method.

A-4.2 **Reagents**

A-4.2.1 *Nitric Acid -- 70* percent.

A-4.2.2 *Sulphuric* **Acid —** *96* percent.

A-4.2.3 *Citric Molybdate Reagent*

Prepared as in A-4.2.3.1 and A-4.2.3.2.

A-4.2.3.1 To *54 g* anhydrous molybdic acid, add 200 ml water and 15 g sodium hydroxide. Heat and stir until dissolved.

A-4.2.3.2 Dissolve 60 g citric acid in 250 to 300 ml water. Add 140 ml concentrated hydrochloric acid (rd 1.18). To this solution, add solution preapared in A-4.2.3.1 with stirring. Filter and dilute to one litre. This solution gives blue-green tinge. Add 0.5 to 1 percent potassium bromate till the colour becomes yellow.

A-4.2.4 *Hydrogen Peroxide — 30* percent (v/v).

A-4.2.5 Mixed *Indicator Solution* — Prepared by mixing 3 volumes of Solution I and 2 volumes of Solution II.

Solution I — Dissolve 0.1 g thymol blue in 2.2 ml of 0.1 N sodium hydroxide and 50 ml alcohol. Dilute to 100 ml with water.

Solution II — Dissolve 0.1 g phenolphthalein in 60 ml alcohol and dilute to 100 ml with water.

A-4.2.6 *Quinoline Solution*

Heat 60 ml concentrated hydrochloric acid in 350 to 400 ml water to 70°C to 80°C. To this hot solution add 50 g quinoline, stir and after cooling, dilute to 1 litre.

A-4.2.7 *Hydrochloric Acid -concentrated* and 0.5 N.

A-4.2.8 *Sodium Hydroxide Solution -0.5 N,*

A-4.3 Procedure

A-4.3.1 Weigh sample to contain 0.5 g quinalphos in a glass ampoule- and introduce it in a 500-ml Kjeldahl digestion flask. Add 20 ml of hydrogen peroxide and cool in ice-cold water. Add 10 ml of sulphuric acid and heat gently under controlled conditions till white fumes of sulphur trioxide come out. Allow to cool, add 15 ml of nitric acid and heat again to disappearance of brown fumes and appearance of white fumes of sulphur trioxide. Allow to cool, add 20 ml of hydrogen peroxide and heat again till white fumes are given off. Cool and make up to 500 ml.

A-4.3.2 To 100 ml aliquot from A-4.3.1, add 50 ml of citric molybdate reagent. Heat to boiling and add very slowly, dropwise, 25 ml of quinoline solution with constant mechanical stirring. Boil for 5 minutes more, cool and filter the precipitate through a Gooch crucible. Wash with water till free from acid. Dissolve the precipitate in a known excess of sodium hydroxide solution (about 50 ml of 0.5 N). Titrate excess alkali with 0.5 N hydrochloric acid using mixed indicator. Carry out a blank determination on the reagents and substract from the volume consumed by the sample.

A-4.4 Calculation

$$\text{Quinalphos content, percent by mass} = \frac{v}{m} \times 2.867$$

where

v = volume in ml of standard sodium hydroxide solution, and

m = mass in g of sample taken for the test.'

(FAD 1)

Printed at Dee Kay Printers. New Delhi-110015, India.